(54) Title: PRODUCING HIGH THROUGHPUT TAPERED STRAIGHT AND CANTILEVERED GLASS STRUCTURES FOR NANODELIVERY AND NANOSENSING

(57) Abstract

Methods for making tapered straight and cantilevered structures and the resulting structures provide maximal nanodelivery of electromagnetic radiation and chemicals and provide nanosensing of ionic phenomena with high efficiency. Glass pulling techniques with specific protocols are applied to glass micropipettes and optical fibers.

Producing High Throughput Tapered Straight and Cantilevered Glass Structures for Nanodelivery and Nanosensing

5   ### 1. Field of the Invention

This invention allows the production of tapered straight and cantilevered structures that are optimized for the maximal nanodelivery of electromagnetic radiation and chemicals and for
10   the maximal sensitivity in the nanosensing of ionic phenomena with high efficiency. These structures are generated so that they can simultaneously act as force sensors with excellent dynamic characteristics. Unlike previous attempts
15   at obtaining such elements with chemical etching techniques our methodology is based solely on the application of glass pulling technologies with specific protocols that can minimize the subwavelength travel of light waves and can
20   maximize the dynamic capabilities of these devices. Unlike the glass etching techniques these glass pulling technologies are universally applicable to glass micropipettes and fibers. In addition optimal geometries of force sensing glass
25   micropipettes are invented for the optimal delivery of chemicals in nanoquantities in a defined and in a nanometrically controlled fashion.

2. Background of the Invention

30   This invention is based on advances in near-field optics.

Near-field optics is the development of optical elements that can work in the near-field of an object that is to be interrogated by light.
35   In essence, the objective is to develop optical

elements that can illuminate, detect and/or enhance optical phenomena within a distance, from the object, that is considerably less than the dimensions of one wavelength. Conventional

5 optical instruments all of which work in the far-field are based on lenses which critically depend on the wave nature of light. Thus, these elements are inherently limited to operation in the far-field with associated problems of

10 diffraction that intrinsically limits resolution to approximately half the wavelength of light. Near-field optical elements not only overcome this diffraction limit of resolution but also relax the wavelength dependence of optical resolution. In

15 its simplest implementation [A. Lewis, M. Isaacson, A. Hartoonian and A. Murray, Biophysical J. 41, 405a (1983); Ultramicroscopy 13, 227 (1984).] near-field optics involves transmitting light through a subwavelength aperture that is

20 brought in close proximity to a surface. The sample or the aperture is then scanned in order to obtain an image or create a pattern with the subwavelength spot of light that emanates from this near-field optical element.

25 3. State of Prior Art

The method of choice that is used throughout the world today to produce near-field optical elements was developed by Lewis et al. [United States Patent Number 4,917,462]. The essence of

30 the methodology of Lewis et al was to use heating, tension and pulling with microprocessor control to produce tapered glass structures that were subsequently coated with metal to form a subwavelength aperture. This was a simple, cheap

and reliable method of producing these subwavelength optical elements. Nonetheless, these elements were produced without much regard to the geometry of the subwavelength tip in terms

5   of reducing the subwavelength region through which the light wave traversed. It is important to realize that the technology in the Lewis et al patent was a general methodology for producing these subwavelength tips through which

10   subwavelength points of light could be produced. This generality can be seen by the fact that the same technology could be used to produce tapered optical elements made out of glass capillaries [A. Harootunian, E. Betzig, M. S. Isaacson and A.

15   Lewis, Appl. Phys. Lett. 49, 674 (1986)] or glass fibers [Betzig E., Trautman J. K., Harris T. D., Weiner J. S. and Kostelak R. L., Science 251, 1468 (1991)]. All of these structures, however, had less than optimized geometries at the tip and thus

20   there were significant evanescent losses that resulted from the transmission of the electromagnetic radiation through the tip.

The first attempts at resolving this problem were made by workers who realized that geometries

25   at the tip that were closer to what would be optimal for high transmission of the light wave through the subwavelength region could be approached with chemical etching of glass fibers [Ohtsu, M. , S. Juang, T. Pangaribuan and M.

30   Kozuma, Proceesing of NFO-1, 131-139 (1993); Jiang, S., Ohsawa, H., Yamada, K., Pangaribuan, T., Ohtsu, M., Imai, K., and Ikai, A., Jpn. J. Appl. Phys. 31, 2282 (1992)] without any previous tapering. Such structures had the potential to

35   generate high throughputs if the coating of these

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structures could be effectively performed. The coating, however, was problematic since the etching technology resulted in damaged surfaces that were difficult to impossible to coat

5 successfully. In addition, the angle required for coating such an etched structure was not compatible with the geometry of such untapered etched fibers. Furthermore, the geometry of such untapered elements (see Figure 1) perturbed

10 significantly the ability of such elements to track rough surfaces. Finally, the untapered nature of these elements made the glass leading to the subwavelength tip very stiff and this crucially reduced the ability of such tips to

15 monitor surface forces. It was this sensitivity of the tapered tips to surface forces [Shalom S., Lieberman K. Lewis A. and Cohen S. R., Rev. Sci. Instrm. 63, 4061 (1992)] that allowed the resolution of one of the principle problems of

20 near-field optics which was the ability to bring such a subwavelength tip close to a surface that was to be interrogated. Also none of the etching methodologies could be applied to tapered micropipette structures whereas the pulling

25 technology is applicable to both tapered micropipettes and optical fibers.

In view of all of these factors it would be best if the standard tapering methodology could be extended to produce a high throughput tip that

30 would also be capable of effectively sensing surface forces.

4. Summary of the Invention

This invention uses pulling technologies as applied to glass tubes to generate without any

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chemical etching a profile that is ideal to produce a high transmission subwavelength optical aperture with very good atomic force sensing capabilities. The same pulling technology with small amendations can be used to optimize similar structures that can combine force sensing capabilities with such applications as the nanodelivery of materials to surfaces.

5. Brief Description of the Drawings

The objects, features and advantages of the present invention will be understood by those of skill in the art from the following detailed description thereof, taken with the accompanying drawings, in which:.

Figure 1 is a diagrammatic illustration of a glass tube tapered in accordance with the present invention;

Figure 2 is a diagrammatic illustration of a tube tapered during a second tapering cycle;

Figure 3 illustrates a mechanism for coating a tapered optical fiber;

Figure 4a illustrates a preferred optical fiber taper for force sensing application;

Figure 4b illustrates a preferred micropipette taper for force sensing applications;

Figure 5 illustrates a cantilevered bent fiber tip;

Figure 6 illustrates a cantilevered pipette constructed in accordance with the invention;

Figure 7 illustrates a cantilevered pipette used for chemistry; and

Figure 8 illustrates a device for applying coating to structures.

6. Description of the Invention

With pulling technology there are many parameters that can be adjusted to obtain the optimal profile.   These are: 1. heat or temperature, 2.   the geometric region that is heated, called the filament, 3.the velocity of the pulling that is measured as the glass softens and begins to separate under a constant load and this velocity is determined by the time of heating, 4. the delay between the start of the pulling and turning off the heating element and 5. the tension of the pulling.   These can all be microprocessor controlled to develop a geometry in which the glass is first tapered to a dimensionality that is above the cut-off for the propagation of electromagnetic radiation in a metallic waveguide (Figure 2.1) and is then cooled before a second tapering cycle is entered in which a cone angle between 30-40° (Figure 2.2) is achieved by multiple pulling cycles. This geometry is shown in Figure 2.   The number of these later cycles depends on the final outer diameter of the tip. In addition to the above these parameters can be altered in order to tailor the devices for other applications such as chemical delivery of materials in nanodimensionalities, optical ion sensing and to optimize the probes for the dynamic movements of the surfaces of the sample initiated by some external stimulus such as light, sound, electricity etc.

5.1   Straight optical elements for maximum throughput of electromagnetic radiation

5.1.1 Tapered Optical Fibers

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5.1.1.1 Pulling

The above pulling technology can be applied with the following parameters for straight optical

fibers in order to get the maximum powers described in this patent.

For the first taper appropriate parameters would be:

Heat	Filament	Velocity	Delay	Pulling
250	3	80	250	10

For the second taper there is a series of three pulls for a 0.1 micron tip with parameters that could be:

Heat	Filament	Velocity	Delay	Pulling
250	0	7	150	10
250	0	10	150	60
290	0	15	126	100

5.1.1.2 Coating

The coating is accomplished with vacuum deposition of metal. One particular example is a thin chrome coating that is then overcoated with a thicker coating of aluminum. For straight elements this coating is accomplished with an angle of 70° between the normal of the optical element and a line drawn between the tip and the filament (see Figure 3). This angle, α, is crucial for the final dimension of the subwavelength aperture at the tip of this second region of taper. The larger the distance between the filament and the tip the more control of the accuracy of the dimension of the aperture. This

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occurs because the cotan a approaches the cos of
this same angle.

5.1.1.3 Force Sensing Capabilities

To optimize the force sensing capabilities of
these tips the initial taper dimension is chosen
to be approximately 20 microns in diameter for
region of 100 microns length just above the second
taper (see Figure 4 a). This is the region in
straight fibers that the flexibility of the
structure is monitored. This dimensionality
controls the resonance frequency and this
frequency, f, can be altered up to 500 kHz with a
width of the resonance, Δf, that can be 0.75 kHz.

5.1.2 Tapered Micropipettes

5.1.2.1 Pulling

Unlike the etching methodologies, the same
pulling technology can be also applied to
micropipettes. Such micropipettes have important
applications in guiding deep ultraviolet light
where optical fibers are not effective. To obtain
the optimal parameters we use the following
protocol:

Heat	Filament	Velocity	Delay	Pulling
390	15	105	200	20

For the second taper there is a series of three
pulls for a 0.1 micron tip with parameters that
could be:

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Heat	Filament	Velocity	Delay	Pulling
220	0	5	200	18
200	0	5	200	15
200	0	30	126	40

5   5.1.2.2 Coating

The coating is accomplished with vacuum deposition of metal. One particular example is a thin chrome coating that is then overcoated with a thicker coating of aluminum. For straight
10   elements this coating is accomplished with an angle of 70° between the normal of the optical element and a line drawn between the tip and the filament (see Figure 3). This angle is crucial for the final dimension of the subwavelength
15   aperture at the tip of this second region of taper. The larger the distance between the filament and the tip the more control of the accuracy of the dimension of the aperture. This occurs because the cotan a approaches the cos of
20   this same angle.

5.1.2.3 Force Sensing Capabilities

To optimize the force sensing capabilities of these tips the initial taper dimension is chosen to be approximately 25 microns in diameter for
25   region of 100 microns length just above the second taper (see Figure 4 b). This is the region in straight tips that the flexibility of the structure is monitored.   This dimensionality controls the resonance frequency and this
30   frequency, f, can be altered up to 60 kHz with a width of the resonance, Δf, that can be 0.3 kHz.

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5.1.3 Cantilevered Fibers

Fibers are bent at the tip according to the procedure that is described by K. Lieberman and A. Lewis, "A Method and a Device for Probe

5　Microscopy" [PCT Application Serial Number PCT/US94/08691]. This bent fiber structure is seen in Figure 5. The angle of the bend in this structure is between 45-60°. The pulling, coating and the resulting resonance frequencies are the

10　same except that the angle of the coating is 85°. In the case of these cantilevered elements such elements can be used for the dynamic sensing of surface movement initiated by an external stimulus such as light, sound, electricity etc.

15　### 5.1.4　Cantilevered Pipettes

The pulling and coating parameters are the same as described above for straight pipettes. However, during the bending operation as described in the Lieberman and Lewis patent application [PCT

20　Application Serial Number PCT/US94/08691] care has to be taken not to block the hollow central core of the pipette. In the case of these cantilevered elements such elements can be used for the dynamic sensing of surface movement initiated by an

25　external stimulus such as light, sound, electricity etc.

5.1.4.1 Cantilevered Pipettes For Light

The structure that is obtained by these pulling parameters is shown in Figure 6. This

30　geometric structure is similar in overall characteristics to what has been obtained with a bent fiber. However, the resonance frequencies

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that can be achieved for this structure are somewhat lower.

5.1.4.2 Cantilevered Pipettes For Chemistry

In this application a liquid or a gas is
5   transmitted through the narrow opening at the tip of the cantilevered micropipette.   Such an uncoated tapered micropipette with force sensing capabilities and a reflective coating only on the cantilever allows the tip to be brought into
10 contact with a surface and this permits the delivery of nanoquantities of material onto the surface.   This causes a chemical change on appropriately chosen surfaces if the nature of the chemical in the tip and the chemical in the
15 surface are reactive one with the other.

In the case of a liquid, under one method of control of the liquid that exists, the tip is accomplished by pressure of the liquid column, by chemical manipulation of the hydrophillicity of
20 the surface and the tip and by the nature of the tip geometry.   Specifically on this last parameter the requirement is exactly opposite to what is required for the light funnels tips discussed above.   When the surface is
25 appropriately wet by the material in the tip the cone angle of the tip can be as small as 7° . This prevents spreading of the liquid column. Measured resonance frequencies of these structures have been measured at 200 kHz.   Additional
30 characteristics that are important in such tips is the need to have high resonance frequencies that will allow for intermittent contact operation and to effectively permit imposed resonances on the tip that will allow, in the case of a liquid

chemical in the tip, for the column of liquid to be effectively and reproducibly broken. Another characteristic difference for chemicals exiting the tip that wet surfaces is the angle of the bend

5   and this angle can reach 90° instead of the 60° that is more characteristic of the light funnels (Fig.7). For other surfaces/tip chemical combinations other angles, tip geometries and resonance frequencies have to be chosen for the

10   most appropriate writing with such tips. Also the force sensing capabilities are of importance in order to give the best sensitivity for measuring the chemical alterations on the surface that have occurred.

15   <u>5.1.4.3 Cantilevered Tapered Micropipettes For Ion Conductance Microscopy</u>
      The pulling and coating parameters are similar to those that are described in the tips that are used for chemical delivery.

20   <u>5.1.4.4 Cantilevered Tapered Micropipettes For Combined Ion Conductance and Optical Ion Sensing</u>
      The pulling and coating parameters are similar to those that are described in the tips that are used for nanodelivery of light since

25   fibers are inserted to illuminate an ion sensing dye filled sol-gel tip from the inside of the micropipette. An additional characteristic is that the tip has to generate sufficient capillary forces to draw a sol-gel into the tip in which

30   there is a dye which changes in optical characteristics with surrounding ion concentration. These sol-gels are conductive to liquid and can be used for both ion conductance

and optical ion sensing.   It is best that such
structures that are to be used in optics are
completely coated with metal as is the case in the
optical funnels.   Materials other than sol-gels
5   can also be used to act as vessels for these dyes.

5.3   NanoSensing of Surface Dynamics

Optimization   of   the   force   sensing
capabilities of the probes described above is
achieved by modulating the dimension of the taper
10   of the glass in the region where the flexibility
of the structure is monitored and this results in
a resonance that is controlled in frequency.   By
controlling this dimension the frequency, f, can
be altered by up to 500 kHz with a width of the
15   resonance, Δf, that can be 0.75 kHz.   This allows
for maximal response of the tips to dynamic
movements of the surface and/or the maximal
sensitivity of the tips for the application of
non-contact protocols in measuring surface forces.

20   ### 5.4   A Device for Appropriate Coating of the Structures Described Above

To achieve appropriate coatings a device was
constructed as shown in Figure 8.   This device is
based on vacuum bell jar and consists of a base
25   8.1,   rotating sealed shaft 8.2, electromotor 8.3,
drive gear wheel 8.4 and set of driven gear wheels
8.5 that are mounted on the periphery of a cup
8.6.   Rotating mounts 8.7 of tapered straight
and/or cantilevered micropipettes and fibers 8.8
30   are mounted on pillars 8.9 and connected with
driven wheel axles by flexible shafts 8.10.
The device operates as following: The motor 8.3
rotates shaft 8.2, which rotates wheel 8.4 and

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wheels 8.5. Furthermore, the rotary motion is transmitted on mounts 8.7 with fibers 8.8 by shafts 8.10. The angle of the tapered glass elements may be changed by a mount that can be

5   raised and lowered and can turn around on the axle 8.11. So that the rotation of the tapered straight or cantilevered micropipette or fiber during the coating process provides uniformity of deposition, and its rotation provides the opportunity to

10   prevent the coating of the tip itself.

6. Advantages Over Prior Art

The techniques described in this patent that can produce high efficiency throughput of light through tapered glass structures with

15   subwavelength apertures at the tip are unique. Previously, all such attempts at producing such structures were based on etching with chemicals. We have achieved high throughputs by inventing a series of pulling protocols that permit very high

20   efficiency transmission of light with excellent capabilities for all force sensing protocols. This has never been achieved before. The throughputs that we have achieved permit the generation of ultrasmall spots of light that could

25   extend to below 20 nm with significant intensities of light in these tips. In addition with small amendations these structures can be altered for excellent delivery of nanoquantities of chemicals with nanometric control of chemistry using the

30   force sensing capabilities of these structures and other uses such as combined force and ion sensing etc.

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7. Applications

A variety of applications are envisioned in
optics, electronics, chemistry, physics and
biology where light of small dimensions is
5   required. Where simultaneous force imaging is
required correlated with optical images these
elements can also be used. In addition the
structures that have been amended slightly to
deliver nanoquantities of chemicals effectively
10   can also be used in a variety of applications
where nanometric control of chemistry is required.

While the invention has been described in
its presently preferred embodiment, it will be
understood that certain modifications can be made
15   in the basic design without departing from the
spirit of the invention as set forth in the
appended claims that is appropriately coated with
metal that includes a tapered region which is
above the cut-off for the transmission of this
20   radiation having a cone angle of 6-10° and a
subsequent tapered section that has a much larger
cone angle that can be as much as 45° such a
structure being optimized for both high throughput
light transmission and excellent capabilities for
25   force sensing as a result of having an ultrahigh
resonance frequencies for example 500 kHz and a Q
factor of 600 and can thus be used in a variety of
force sensing protocols. section that has a much
larger cone angle that can be as much as 45° such
30   a structure being optimized for both high
throughput light transmission and excellent
capabilities for force sensing as a result of
having an ultrahigh resonance frequencies for
example 500 kHz and a Q factor of 600 and can thus
35   be used in a variety of force sensing protocols.

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WHAT IS CLAIMED IS:

1.   A device comprised of a tapered glass structure produced from glass tubes by pulling technologies to generate an optimized geometry for high transmission efficiency of electromagnetic radiation.

2.   A device as in claim 1 that is made out of a glass capillary.

3.   A device as in claim 1 that is made out of an optical fiber.

4.   A device as in claim 2 that is bent near the tip to produce a cantilevered structure suitable for normal force sensing in a variety of attractive, repulsive and non-contact imaging modes

5.   A device as in claim 3 that is bent near the tip to produce a cantilevered structure suitable for normal force sensing in a variety of attractive, repulsive and non-contact imaging modes

6.   A device as in claim 2 that is cantilevered and optimized for delivery of nanoquantities of liquid in a controlled fashion so that chemistry with nanometric control can be performed and this is accomplished by pressure on the liquid column inside the tapered micropipette , by chemical manipulation of the hydrophillicity of the surface and the tip, by the nature of the tapered geometry of the tip and the angle of the cantilever relative to the tip in order to prevent

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the spreading of the liquid column on the surface
on which nanochemistry is to be performed

7.   A device as in claim 6 with resonance
frequencies that will allow for intermittent
contact operation and will effectively permit
imposed resonances on the tip that will allow, in
the case of a liquid chemical in the tip, for the
column of liquid to be effectively and
reproducibly broken

8.   A device as in claim 6 that is also
optimized for maximum force sensitivity

9.   A device. as in claim 7 that is also
optimized for maximum force sensitivity

10.   A device as in claim 2 that is
cantilevered and optimized for ion conductance.

11. A device as in claim 10 that optimizes
the transmission of light that can excite either
directly or through an optical fiber threaded
through the micropipette an ion sensing dye in a
sol-gel glass at the tip of the micropipette that
is also simultaneously conductive to ions and is
also capable of static and dynamic force sensing

12. A device for the coating of tapered
straight and cantilevered structures so that thin
films can be deposited on these glass structures
by rotation about the axis of the straight glass
tip and/or by accurately varying the angle of
incidence between the source and the tip target

13.   A device for the coating of tapered straight and cantilevered structures for maximal throughput of electromagnetic radiation by controlling the distance accurately between the

5   point of evaporation and the tip of the straight micropipette or fiber or the cantilever of the bent micropipette or fiber and this distance control is related to the diameter of the central rotator of all the tips so that the cotan of the

10   angle between the axis of the straight micropipette or fiber or the cantilever of the bent micropipette or fiber approaches the cos of this same angle.

14.   A device for the coating of tapered

15   straight and cantilevered structures for maximal throughput of electromagnetic radiation by controlling the angle as in claim 12 together with the distance as in claim 13 to give most accurate control of the final dimension of the resulting

20   aperture at the tip and controlling the thickness of the coating for maximum throughput of electromagnetic radiation

15.   A device in which the force sensing capabilities of the probes as described in claim

25   1 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can

30   be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface

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and/or application of non-contact protocols in measuring surface forces

16. A device in which the force sensing capabilities of the probes as described in claim 2 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

17. A device in which the force sensing capabilities of the probes as described in claim 3 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

18. A device in which the force sensing capabilities of the probes as described in claim 4 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region

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where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500

5   kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

10   19. A device in which the force sensing capabilities of the probes as described in claim 5 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is

15   monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response

20   of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

         20. A device in which the force sensing capabilities of the probes as described in claim

25   6 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can

30   be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface

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and/or application of non-contact protocols in measuring surface forces

21. A device in which the force sensing capabilities of the probes as described in claim 7 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application. of non-contact protocols in measuring surface forces

22. A device in which the force sensing capabilities of the probes as described in claim 8 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

23. A device in which the force sensing capabilities of the probes as described in claim 9 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region

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where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500

5   kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

10   24.   A device in which the force sensing capabilities of the probes as described in claim 10 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is

15   monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response

20   of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

          25.   A device in which the force sensing capabilities of the probes as described in claim

25   11 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can

30   be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface

and/or application of non-contact protocols in measuring surface forces.

26. A method of tapering a glass structure produced from glass tubes by glass pulling technologies to generate an optimized geometry for high transmission efficiency of electromagnetic radiation that is appropriately coated with metal that includes a tapered region which is above the cut-off for the transmission of this radiation having a cone angle of 6-10° and a subsequent tapered section that has a much larger cone angle that can be as much as 45° such a structure being optimized for both high throughput light transmission and excellent capabilities for force sensing as a result of having an ultrahigh resonance frequencies for example 500 kHz and a Q factor of 600 and can thus be used in a variety of force sensing protocols.

27. A method as in claim 26 using a glass capillary

28. A method as in claim 26 using an optical fiber

29. A method as in claim 27 that produces a cantilevered structure by bending the tip so that the structure is suitable for normal force sensing in a variety of attractive, repulsive and non-contact imaging modes.

30. A device as in claim 28 that produces a cantilevered structure by bending the tip so that the structure is suitable for normal force sensing

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in a variety of attractive, repulsive and non-contact imaging modes.

31. A method as in claim 27 that produces a cantilevered structure optimized for delivery of
5   nanoquantities of liquid in a controlled fashion so that chemistry with nanometric control can be performed and this is accomplished by pressure on the liquid column inside the tapered micropipette, by chemical manipulation of the hydrophillicity of
10 the surface and the tip, by the nature of the tapered geometry of the tip and the angle of the cantilever relative to the tip in order to prevent the spreading of the liquid column on the surface on which nanochemistry is to be performed .

15 32. A method as in claim 31 for producing a structure that has resonance frequencies that will allow for intermittent contact operation and will effectively permit imposed resonances on the tip that will allow, in the case of a liquid chemical
20 in the tip, for the column of liquid to be effectively and reproducibly broken.

33. A method as in claim 31 that also optimizes the structure for maximum force sensitivity.

25 34. A method as in claim 32 that also optimizes the structure for maximum force sensitivity.

35. A method as in claim 27 for producing a cantilevered structure optimized for ion
30 conductance.

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36. A method as in claim 35 for producing a structure that optimizes the transmission of light that can excite either directly or through an optical fiber threaded through the micropipette an ion sensing dye at the tip of the micropipette in a sol-gel glass that is also simultaneously conductive to ions and is also capable of static and dynamic force sensing.

37. A method for the coating of tapered straight and cantilevered structures so that thin films can be deposited on these glass structures by rotation about the axis of the straight glass tip and/or by accurately varying the angle of incidence between the source and the tip target.

38. A method for the coating of tapered straight and cantilevered structures for maximal throughput of electromagnetic radiation by controlling the distance accurately between the point of evaporation and the tip of the straight micropipette or fiber or the cantilever of the bent micropipette or fiber and this distance control is related to the diameter of the central rotator of all the tips so that the cotan of the angle between the axis of the straight micropipette or fiber or the cantilever of the bent micropipette or fiber approaches the cos of this same angle.

38. A method for the coating of tapered straight and cantilevered structures for maximal throughput of electromagnetic radiation by controlling the angle as in claim 37 together with the distance as in claim 38 to give most accurate

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control of the final dimension of the resulting
aperture at the tip and controlling the thickness
of the coating for maximum throughput of
electromagnetic radiation.

5   40.   A method for the optimization of the
force sensing capabilities of the probes as
described in claim 26 so that the dimension of the
taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
10   monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
15   of the tips to dynamic movements of the surface
and/or application of non-contact protocols in
measuring surface forces.

           41.   A method for the optimization of the
force sensing capabilities of the probes as
20   described in claim 28 so that the dimension of the
taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
25   be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
of the tips to dynamic movements of the surface
and/or application of non-contact protocols in
30   measuring surface forces.

           42.   A method for optimization of the force
sensing capabilities of the probes as described in

27

claim 29 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

43. A method for optimization of the force sensing capabilities of the probes as described in claim 30 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

44. A method for the optimization of the force sensing capabilities of the probes as described in claim 31 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be

0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

5   45.   A method for optimization of the force sensing capabilities of the probes as described in claim 32 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and

10   this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the

15   tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

       46.   A method for the optimization of the force sensing capabilities of the probes as

20   described in claim 33 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can

25   be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in

30   measuring surface forces.

       47.   A method for the optimization of the force sensing capabilities of the probes as
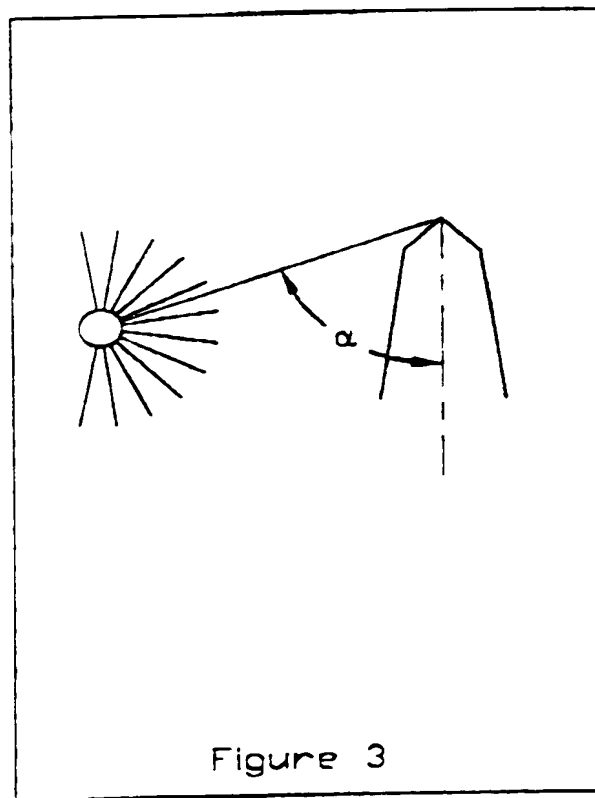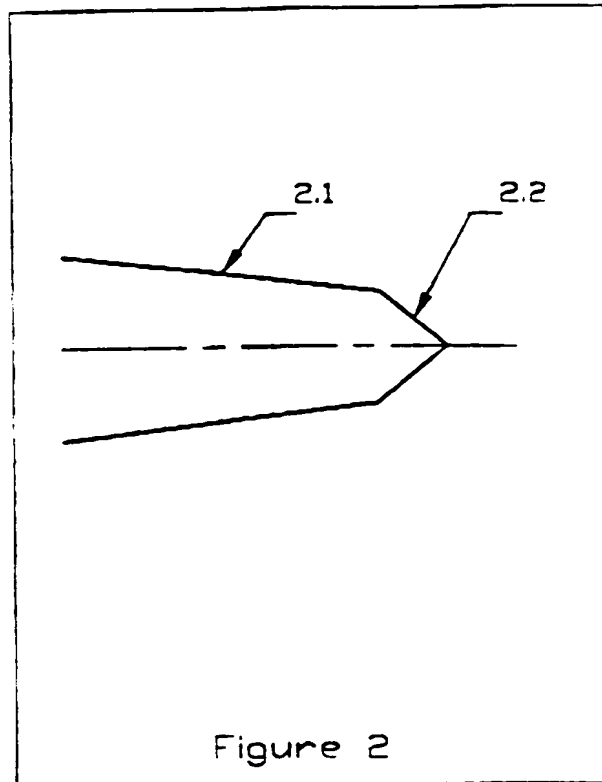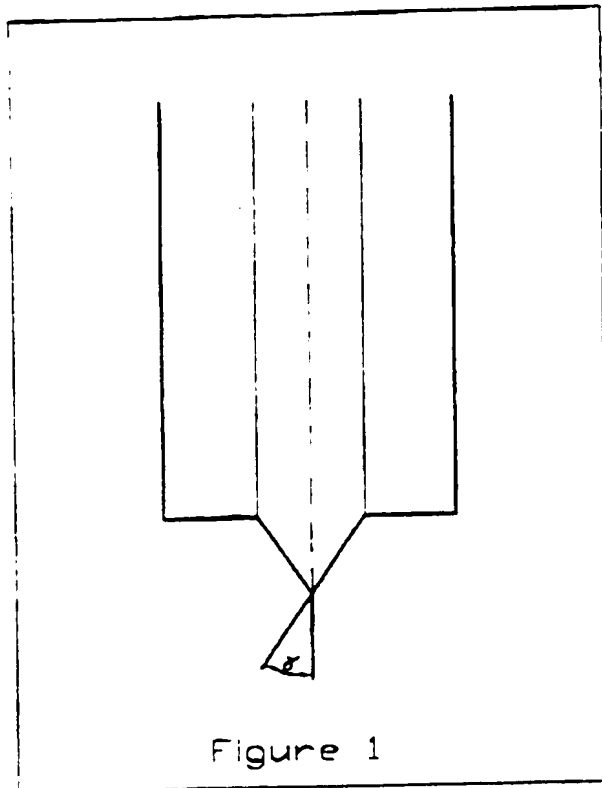
described in claim 34 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.
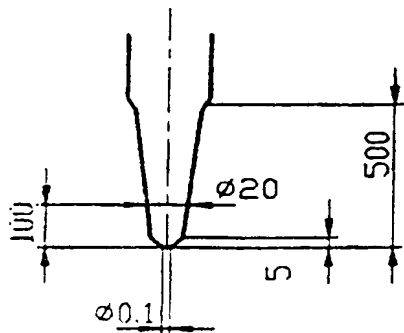
48. A method for the optimization of the force sensing capabilities of the probes as described in claim 35 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

49. A method for the optimization of the force sensing capabilities of the probes as described in claim 36 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be

0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.
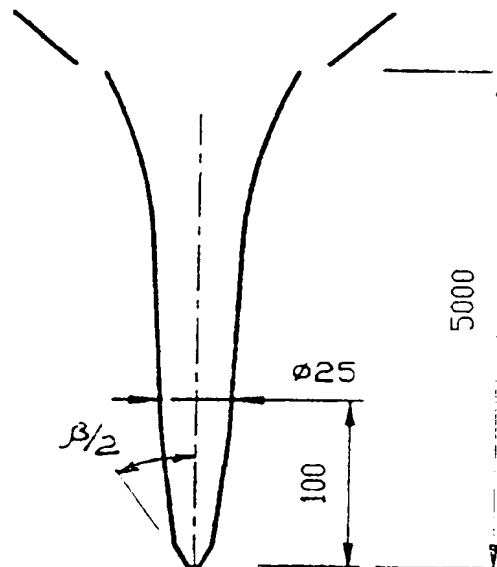
5   50.   A method for the optimization of the force sensing capabilities of the probes as described in claim 37 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is

10   monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response

15   of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

Figure 1

Figure 2

Figure 3
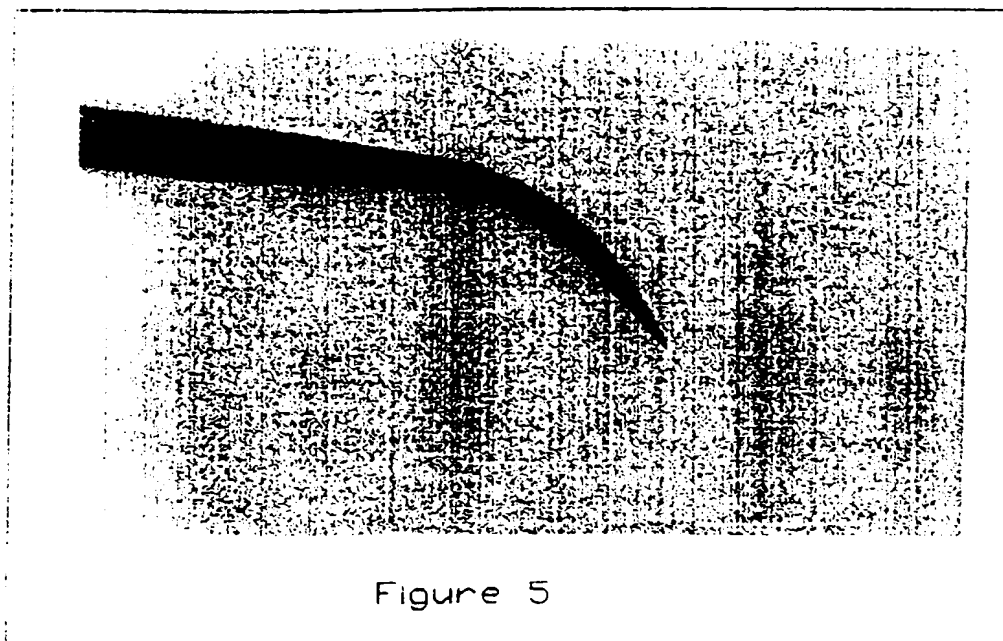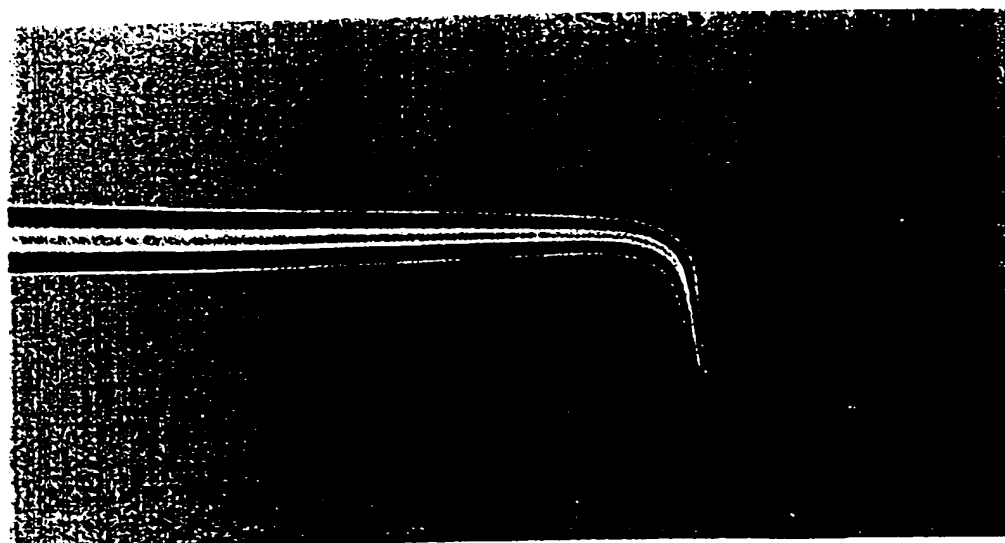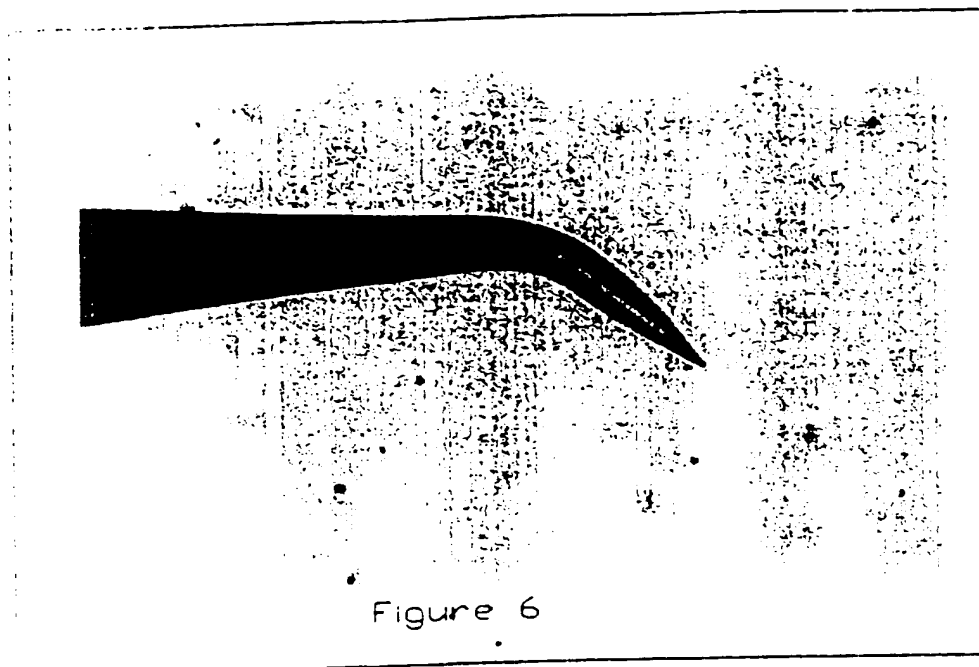
Figure 4a

All dimensions are in micrometers

Figure 4b
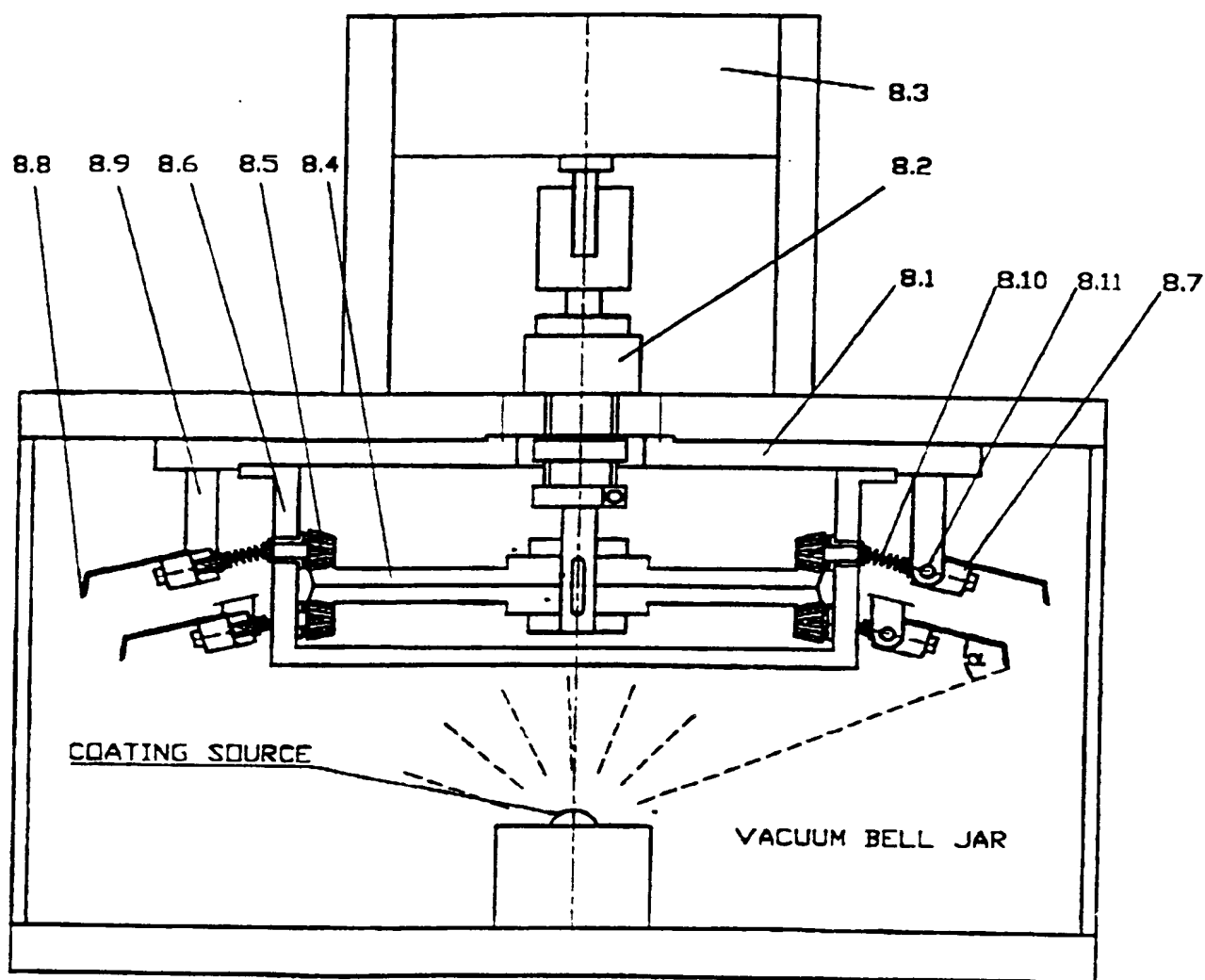
All dimensions are in micrometers

Figure 5

Figure 6

Figure 7

COATING SOURCE

VACUUM BELL JAR

Figure 8

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(51) International Patent Classification [6] : **G02B 6/26**	**A3**	**(11) International Publication Number:** **WO 98/37440**
		(43) International Publication Date: 27 August 1998 (27.08.98)

(21) International Application Number: PCT/US98/01577

(22) International Filing Date: 9 February 1998 (09.02.98)

(30) Priority Data:
120181   9 February 1997 (09.02.97)   IL

(71)(72) Applicant and Inventor: LEWIS, Aaron [US/IL]; Neve Shaanan Street18/14, 93708 Jerusalem (IL).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: FISH, Galina [IL/IL]; 166, 90916 Ishuv Nokdim (IL). KOKOTOV, Sophia [IL/IL]; Avnei HaHoshen Street60/4, 98450 Maale Adumim (IL). KHACHAIRYAN, Edward [IL/IL]; Shtern Street63/73, 96750 Jerusalem (IL). IGNATOV, Andrey [IL/IL]; Herzel Street52/9, 96141 Jerusalem (IL). GLAZER, Rimma [IL/IL]; HaNurit Street127/17, Jerusalem (IL). KOMISSAR, Anatoly [IL/IL]; Brazil Street102/17, 96784 Jerusalem (IL). HEIFEZ, Yuri [IL/IL]; Avivit Street122/2, 96507 Jerusalem (IL). STRINKOVSKY, Alina [IL/IL]; Keren HaYesod Street1115/21, 99032 Beit Shemesh (IL). LIEBERMAN, Klony [IL/IL]; Shai Agnon Street14/15, Jerusalem (IL).

(74) Agents: COOPER, George, M. et al.; Jones, Tullar & Cooper, P.C., P.O. Box 2266, Eads Station, Arlington, VA 22202 (US).

(81) Designated States: JP, US, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published
With international search report.
Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.

(88) Date of publication of the international search report:
8 October 1998 (08.10.98)

(54) Title: GLASS STRUCTURES FOR NANODELIVERY AND NANOSENSING

(57) Abstract

Methods for making tapered straight and cantilevered structures and the resulting structures provide maximal nanodelivery of electromagnetic radiation and chemicals and provide nanosensing of ionic phenomena with high efficiency. Glass pulling techniques with specific protocols are applied to glass micropipettes and optical fibers.

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INTERNATIONAL SEARCH REPORT

A.   CLASSIFICATION OF SUBJECT MATTER

IPC(6)   :G02B 6/26

US CL   :385/43, 147, 38, 12

According to International Patent Classification (IPC) or to both national classification and IPC

B.   FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. :   385/43, 147, 38, 12, 15.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

USPTO data base APS. Search terms: glass, tube, optical fiber, tapered, sensing.

C.   DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,877,300 A (NEWHOUSE ET AL) 31 OCTOBER 1989 (31/10/89), see entire document.	1-50
A	US 4,915,467 A (BERKEY) 10 APRIL 1990 (10/04/90), see entire document.	1-50
A	US 5,044,716 A (BERKEY) 03 SEPTEMBER 1991 (03/09/91), see entire document.	1-50

☐ Further documents are listed in the continuation of Box C.   ☐ See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
20 MAY 1998	1 7 AUG 1998

Name and mailing address of the ISA/US	Authorized officer
Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	JOHN NGO
Facsimile No. (703) 305-3230	Telephone No (703) 308-0297

Form PCT ISA 210 (second sheet)(July 1992)★

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(54) Title: GLASS STRUCTURES FOR NANODELIVERY AND NANOSENSING

(57) Abstract

Methods for making tapered straight and cantilevered structures and the resulting structures provide maximal nanodelivery of electromagnetic radiation and chemicals and provide nanosensing of ionic phenomena with high efficiency. Glass pulling techniques with specific protocols are applied to glass micropipettes and optical fibers.

GLASS STRUCTURES FOR NANODELIVERY AND NANOSENSING

5   1. Field of the Invention

This invention allows the production of tapered straight and cantilevered structures that are optimized for the maximal nanodelivery of electromagnetic radiation and chemicals and for
10   the maximal sensitivity in the nanosensing of ionic phenomena with high efficiency. These structures are generated so that they can simultaneously act as force sensors with excellent dynamic characteristics. Unlike previous attempts
15   at obtaining such elements with chemical etching techniques our methodology is based solely on the application of glass pulling technologies with specific protocols that can minimize the subwavelength travel of light waves and can
20   maximize the dynamic capabilities of these devices. Unlike the glass etching techniques these glass pulling technologies are universally applicable to glass micropipettes and fibers. In addition optimal geometries of force sensing glass
25   micropipettes are invented for the optimal delivery of chemicals in nanoquantities in a defined and in a nanometrically controlled fashion.

2. Background of the Invention
30   This invention is based on advances in near-field optics.

Near-field optics is the development of optical elements that can work in the near-field of an object that is to be interrogated by light.
35   In essence, the objective is to develop optical

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elements that can illuminate, detect and/or enhance optical phenomena within a distance, from the object, that is considerably less than the dimensions of one wavelength. Conventional

5   optical instruments all of which work in the far-field are based on lenses which critically depend on the wave nature of light. Thus, these elements are inherently limited to operation in the far-field with associated problems of

10   diffraction that intrinsically limits resolution to approximately half the wavelength of light. Near-field optical elements not only overcome this diffraction limit of resolution but also relax the wavelength dependence of optical resolution. In

15   its simplest implementation [A. Lewis, M. Isaacson, A. Hartoonian and A. Murray, Biophysical J. 41, 405a (1983); Ultramicroscopy 13, 227 (1984).] near-field optics involves transmitting light through a subwavelength aperture that is

20   brought in close proximity to a surface. The sample or the aperture is then scanned in order to obtain an image or create a pattern with the subwavelength spot of light that emanates from this near-field optical element.

25   3. State of Prior Art
          The method of choice that is used throughout the world today to produce near-field optical elements was developed by Lewis et al. [United States Patent Number 4,917,462]. The essence of

30   the methodology of Lewis et al was to use heating, tension and pulling with microprocessor control to produce tapered glass structures that were subsequently coated with metal to form a subwavelength aperture. This was a simple, cheap

and reliable method of producing these subwavelength optical elements. Nonetheless, these elements were produced without much regard to the geometry of the subwavelength tip in terms

5   of reducing the subwavelength region through which the light wave traversed. It is important to realize that the technology in the Lewis et al patent was a general methodology for producing these subwavelength tips through which

10   subwavelength points of light could be produced. This generality can be seen by the fact that the same technology could be used to produce tapered optical elements made out of glass capillaries [A. Harootunian, E. Betzig, M. S. Isaacson and A.

15   Lewis, Appl. Phys. Lett. 49, 674 (1986)] or glass fibers [Betzig E., Trautman J. K., Harris T. D., Weiner J. S. and Kostelak R. L., Science 251, 1468 (1991)]. All of these structures, however, had less than optimized geometries at the tip and thus

20   there were significant evanescent losses that resulted from the transmission of the electromagnetic radiation through the tip.

     The first attempts at resolving this problem were made by workers who realized that geometries

25   at the tip that were closer to what would be optimal for high transmission of the light wave through the subwavelength region could be approached with chemical etching of glass fibers [Ohtsu, M. , S. Juang, T. Pangaribuan and M.

30   Kozuma, Proceesing of NFO-1, 131-139 (1993); Jiang, S., Ohsawa, H., Yamada, K., Pangaribuan, T., Ohtsu, M., Imai, K., and Ikai, A., Jpn. J. Appl. Phys. 31, 2282 (1992)] without any previous tapering. Such structures had the potential to

35   generate high throughputs if the coating of these

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structures could be effectively performed. The
coating, however, was problematic since the
etching technology resulted in damaged surfaces
that were difficult to impossible to coat
5   successfully. In addition, the angle required for
coating such an etched structure was not
compatible with the geometry of such untapered
etched fibers. Furthermore, the geometry of such
untapered elements (see Figure 1) perturbed
10   significantly the ability of such elements to
track rough surfaces. Finally, the untapered
nature of these elements made the glass leading to
the subwavelength tip very stiff and this
crucially reduced the ability of such tips to
15   monitor surface forces. It was this sensitivity
of the tapered tips to surface forces [Shalom S.,
Lieberman K. Lewis A. and Cohen S. R., Rev. Sci.
Instrm. 63, 4061 (1992)] that allowed the
resolution of one of the principle problems of
20   near-field optics which was the ability to bring
such a subwavelength tip close to a surface that
was to be interrogated. Also none of the etching
methodologies could be applied to tapered
micropipette structures whereas the pulling
25   technology is applicable to both tapered
micropipettes and optical fibers.

In view of all of these factors it would be
best if the standard tapering methodology could be
extended to produce a high throughput tip that
30   would also be capable of effectively sensing
surface forces.

4. Summary of the Invention

This invention uses pulling technologies as
applied to glass tubes to generate without any

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chemical etching a profile that is ideal to produce a high transmission subwavelength optical aperture with very good atomic force sensing capabilities. The same pulling technology with small amendations can be used to optimize similar structures that can combine force sensing capabilities with such applications as the nanodelivery of materials to surfaces.

5. Brief Description of the Drawings

The objects, features and advantages of the present invention will be understood by those of skill in the art from the following detailed description thereof, taken with the accompanying drawings, in which:.

Figure 1 is a diagrammatic illustration of a glass tube tapered in accordance with the present invention;

Figure 2 is a diagrammatic illustration of a tube tapered during a second tapering cycle;

Figure 3 illustrates a mechanism for coating a tapered optical fiber;

Figure 4a illustrates a preferred optical fiber taper for force sensing application;

Figure 4b illustrates a preferred micropipette taper for force sensing applications;

Figure 5 illustrates a cantilevered bent fiber tip;

Figure 6 illustrates a cantilevered pipette constructed in accordance with the invention;

Figure 7 illustrates a cantilevered pipette used for chemistry; and

Figure 8 illustrates a device for applying coating to structures.

6. Description of the Invention

With pulling technology there are many parameters that can be adjusted to obtain the optimal profile. These are: 1. heat or temperature, 2. the geometric region that is heated, called the filament, 3.the velocity of the pulling that is measured as the glass softens and begins to separate under a constant load and this velocity is determined by the time of heating, 4. the delay between the start of the pulling and turning off the heating element and 5. the tension of the pulling. These can all be microprocessor controlled to develop a geometry in which the glass is first tapered to a dimensionality that is above the cut-off for the propagation of electromagnetic radiation in a metallic waveguide (Figure 2.1) and is then cooled before a second tapering cycle is entered in which a cone angle between 30-40° (Figure 2.2) is achieved by multiple pulling cycles. This geometry is shown in Figure 2. The number of these later cycles depends on the final outer diameter of the tip. In addition to the above these parameters can be altered in order to tailor the devices for other applications such as chemical delivery of materials in nanodimensionalities, optical ion sensing and to optimize the probes for the dynamic movements of the surfaces of the sample initiated by some external stimulus such as light, sound, electricity etc.

5.1 Straight optical elements for maximum throughput of electromagnetic radiation

5.1.1 Tapered Optical Fibers

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5.1.1.1   Pulling

The above pulling technology can be applied
with the following parameters for straight optical

5   fibers in order to get the maximum powers
described in this patent.
For the first taper appropriate parameters would
be:

Heat	Filament	Velocity	Delay	Pulling
250	3	80	250	10

10   For the second taper there is a series of three
pulls for a 0.1 micron tip with parameters that
could be:

Heat	Filament	Velocity	Delay	Pulling
250	0	7	150	10
250	0	10	150	60
290	0	15	126	100

5.1.1.2   Coating

The coating is accomplished with vacuum
deposition of metal. One particular example is a
20   thin chrome coating that is then overcoated with
a thicker coating of aluminum.   For straight
elements this coating is accomplished with an
angle of 70° between the normal of the optical
element and a line drawn between the tip and the
25   filament (see Figure 3).   This angle, α, is
crucial for the final dimension of the
subwavelength aperture at the tip of this second
region of taper. The larger the distance between
the filament and the tip the more control of the
30   accuracy of the dimension of the aperture.   This

occurs because the cotan a approaches the cos of this same angle.

5.1.1.3   Force Sensing Capabilities

To optimize the force sensing capabilities of these tips the initial taper dimension is chosen to be approximately 20 microns in diameter for region of 100 microns length just above the second taper (see Figure 4 a). This is the region in straight fibers that the flexibility of the structure is monitored. This dimensionality controls the resonance frequency and this frequency, f, can be altered up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz.

5.1.2   Tapered Micropipettes

5.1.2.1   Pulling

Unlike the etching methodologies, the same pulling technology can be also applied to micropipettes. Such micropipettes have important applications in guiding deep ultraviolet light where optical fibers are not effective. To obtain the optimal parameters we use the following protocol:

Heat	Filament	Velocity	Delay	Pulling
390	15	105	200	20

For the second taper there is a series of three pulls for a 0.1 micron tip with parameters that could be:

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Heat	Filament	Velocity	Delay	Pulling
220	0	5	200	18
200	0	5	200	15
200	0	30	126	40

5   <u>5.1.2.2 Coating</u>

The coating is accomplished with vacuum deposition of metal. One particular example is a thin chrome coating that is then overcoated with a th?cker coating of aluminum. For straight

10 elements this coating is accomplished with an angle of 70° between the normal of the optical element and a line drawn between the tip and the filament (see Figure 3). This angle is crucial for the final dimension of the subwavelength

15 aperture at the tip of this second region of taper. The larger the distance between the filament and the tip the more control of the accuracy of the dimension of the aperture. This occurs because the cotan a approaches the cos of

20 this same angle.

<u>5.1.2.3 Force Sensing Capabilities</u>

To optimize the force sensing capabilities of these tips the initial taper dimension is chosen to be approximately 25 microns in diameter for

25 region of 100 microns length just above the second taper (see Figure 4 b). This is the region in straight tips that the flexibility of the structure is monitored.   This dimensionality controls the resonance frequency and this

30 frequency, f, can be altered up to 60 kHz with a width of the resonance, Δf, that can be 0.3 kHz.

10

5.1.3 Cantilevered Fibers

Fibers are bent at the tip according to the procedure that is described by K. Lieberman and A. Lewis, "A Method and a Device for Probe Microscopy" [PCT Application Serial Number PCT/US94/08691]. This bent fiber structure is seen in Figure 5. The angle of the bend in this structure is between 45-60°. The pulling, coating and the resulting resonance frequencies are the same except that the angle of the coating is 85°. In the case of these cantilevered elements such elements can be used for the dynamic sensing of surface movement initiated by an external stimulus such as light, sound, electricity etc.

5.1.4 Cantilevered Pipettes

The pulling and coating parameters are the same as described above for straight pipettes. However, during the bending operation as described in the Lieberman and Lewis patent application [PCT Application Serial Number PCT/US94/08691] care has to be taken not to block the hollow central core of the pipette. In the case of these cantilevered elements such elements can be used for the dynamic sensing of surface movement initiated by an external stimulus such as light, sound, electricity etc.

5.1.4.1 Cantilevered Pipettes For Light

The structure that is obtained by these pulling parameters is shown in Figure 6. This geometric structure is similar in overall characteristics to what has been obtained with a bent fiber. However, the resonance frequencies

that can be achieved for this structure are somewhat lower.

5.1.4.2 Cantilevered Pipettes For Chemistry

In this application a liquid or a gas is transmitted through the narrow opening at the tip of the cantilevered micropipette. Such an uncoated tapered micropipette with force sensing capabilities and a reflective coating only on the cantilever allows the tip to be brought into contact with a surface and this permits the delivery of nanoquantities of material onto the surface. This causes a chemical change on appropriately chosen surfaces if the nature of the chemical in the tip and the chemical in the surface are reactive one with the other.

In the case of a liquid, under one method of control of the liquid that exists, the tip is accomplished by pressure of the liquid column, by chemical manipulation of the hydrophillicity of the surface and the tip and by the nature of the tip geometry. Specifically on this last parameter the requirement is exactly opposite to what is required for the light funnels tips discussed above. When the surface is appropriately wet by the material in the tip the cone angle of the tip can be as small as 7° . This prevents spreading of the liquid column. Measured resonance frequencies of these structures have been measured at 200 kHz. Additional characteristics that are important in such tips is the need to have high resonance frequencies that will allow for intermittent contact operation and to effectively permit imposed resonances on the tip that will allow, in the case of a liquid

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chemical in the tip, for the column of liquid to be effectively and reproducibly broken.   Another characteristic difference for chemicals exiting the tip that wet surfaces is the angle of the bend

5   and this angle can reach 90° instead of the 60° that is more characteristic of the light funnels (Fig.7).   For   other   surfaces/tip   chemical combinations   other   angles,   tip   geometries   and resonance frequencies have to be chosen for the

10   most appropriate writing with such tips. Also the force sensing capabilities are of importance in order to give the best sensitivity for measuring the chemical alterations on the surface that have occurred.

15   5.1.4.3 Cantilevered Tapered Micropipettes For Ion Conductance Microscopy
         The   pulling   and   coating   parameters   are similar to those that are described in the tips that are used for chemical delivery.

20   5.1.4.4   Cantilevered Tapered Micropipettes For Combined Ion Conductance and Optical Ion Sensing
         The   pulling   and   coating   parameters   are similar to those that are described in the tips that are used for nanodelivery of light since

25   fibers are inserted to illuminate an ion sensing dye filled sol-gel tip from the inside of the micropipette.   An additional characteristic is that the tip has to generate sufficient capillary forces to draw a sol-gel into the tip in which

30   there   is   a   dye   which   changes   in   optical characteristics   with   surrounding   ion concentration.   These sol-gels are conductive to liquid and can be used for both ion conductance

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and optical ion sensing. It is best that such
structures that are to be used in optics are
completely coated with metal as is the case in the
optical funnels. Materials other than sol-gels

5   can also be used to act as vessels for these dyes.

5.3 NanoSensing of Surface Dynamics
       Optimization   of   the   force   sensing
capabilities of the probes described above is
achieved by modulating the dimension of the taper

10   of the glass in the region where the flexibility
of the structure is monitored and this results in
a resonance that is controlled in frequency. By
controlling this dimension the frequency, f, can
be altered by up to 500 kHz with a width of the

15   resonance, Δf, that can be 0.75 kHz. This allows
for maximal response of the tips to dynamic
movements of the surface and/or the maximal
sensitivity of the tips for the application of
non-contact protocols in measuring surface forces.

20   5.4   A Device for Appropriate Coating of the
Structures Described Above
       To achieve appropriate coatings a device was
constructed as shown in Figure 8. This device is
based on vacuum bell jar and consists of a base

25   8.1, rotating sealed shaft 8.2, electromotor 8.3,
drive gear wheel 8.4 and set of driven gear wheels
8.5 that are mounted on the periphery of a cup
8.6. Rotating mounts 8.7 of tapered straight
and/or cantilevered micropipettes and fibers 8.8

30   are mounted on pillars 8.9 and connected with
driven wheel axles by flexible shafts 8.10.
       The device operates as following: The motor 8.3
rotates shaft 8.2, which rotates wheel 8.4 and

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wheels 8.5. Furthermore, the rotary motion is transmitted on mounts 8.7 with fibers 8.8 by shafts 8.10. The angle of the tapered glass elements may be changed by a mount that can be

5   raised and lowered and can turn around on the axle 8.11. So that the rotation of the tapered straight or cantilevered micropipette or fiber during the coating process provides uniformity of deposition, and its rotation provides the opportunity to

10   prevent the coating of the tip itself.

6. Advantages Over Prior Art

       The techniques described in this patent that can produce high efficiency throughput of light through tapered glass structures with

15   subwavelength apertures at the tip are unique. Previously, all such attempts at producing such structures were based on etching with chemicals. We have achieved high throughputs by inventing a series of pulling protocols that permit very high

20   efficiency transmission of light with excellent capabilities for all force sensing protocols. This has never been achieved before. The throughputs that we have achieved permit the generation of ultrasmall spots of light that could

25   extend to below 20 nm with significant intensities of light in these tips. In addition with small amendations these structures can be altered for excellent delivery of nanoquantities of chemicals with nanometric control of chemistry using the

30   force sensing capabilities of these structures and other uses such as combined force and ion sensing etc.

7. Applications

     A variety of applications are envisioned in
optics, electronics, chemistry, physics and
biology where light of small dimensions is
5   required. Where simultaneous force imaging is
required correlated with optical images these
elements can also be used. In addition the
structures that have been amended slightly to
deliver nanoquantities of chemicals effectively
10   can also be used in a variety of applications
where nanometric control of chemistry is required.

     While the invention has been described in
its presently preferred embodiment, it will be
understood that certain modifications can be made
15   in the basic design without departing from the
spirit of the invention as set forth in the
appended claims that is appropriately coated with
metal that includes a tapered region which is
above the cut-off for the transmission of this
20   radiation having a cone angle of 6-10° and a
subsequent tapered section that has a much larger
cone angle that can be as much as 45° such a
structure being optimized for both high throughput
light transmission and excellent capabilities for
25   force sensing as a result of having an ultrahigh
resonance frequencies for example 500 kHz and a Q
factor of 600 and can thus be used in a variety of
force sensing protocols. section that has a much
larger cone angle that can be as much as 45° such
30   a structure being optimized for both high
throughput light transmission and excellent
capabilities for force sensing as a result of
having an ultrahigh resonance frequencies for
example 500 kHz and a Q factor of 600 and can thus
35   be used in a variety of force sensing protocols.

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WHAT IS CLAIMED IS:

1.   A device comprised of a tapered glass
structure produced from glass tubes by pulling
technologies to generate an optimized geometry for
5   high transmission efficiency of electromagnetic
radiation.

2.   A device as in claim 1 that is made out
of a glass capillary.

3.   A device as in claim 1 that is made out
10   of an optical fiber.

4.   A device as in claim 2 that is bent near
the tip to produce a cantilevered structure
suitable for normal force sensing in a variety of
attractive, repulsive and non-contact imaging
15   modes

5.   A device as in claim 3 that is bent near
the tip to produce a cantilevered structure
suitable for normal force sensing in a variety of
attractive, repulsive and non-contact imaging
20   modes

6.   A device as in claim 2 that is
cantilevered and optimized for delivery of
nanoquantities of liquid in a controlled fashion
so that chemistry with nanometric control can be
25   performed and this is accomplished by pressure on
the liquid column inside the tapered micropipette
, by chemical manipulation of the hydrophillicity
of the surface and the tip, by the nature of the
tapered geometry of the tip and the angle of the
30   cantilever relative to the tip in order to prevent

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the spreading of the liquid column on the surface on which nanochemistry is to be performed

7. A device as in claim 6 with resonance frequencies that will allow for intermittent contact operation and will effectively permit imposed resonances on the tip that will allow, in the case of a liquid chemical in the tip, for the column of liquid to be effectively and reproducibly broken

8. A device as in claim 6 that is also optimized for maximum force sensitivity

9. A device. as in claim 7 that is also optimized for maximum force sensitivity

10. A device as in claim 2 that is cantilevered and optimized for ion conductance.

11. A device as in claim 10 that optimizes the transmission of light that can excite either directly or through an optical fiber threaded through the micropipette an ion sensing dye in a sol-gel glass at the tip of the micropipette that is also simultaneously conductive to ions and is also capable of static and dynamic force sensing

12. A device for the coating of tapered straight and cantilevered structures so that thin films can be deposited on these glass structures by rotation about the axis of the straight glass tip and/or by accurately varying the angle of incidence between the source and the tip target

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13.   A device for the coating of tapered
straight and cantilevered structures for maximal
throughput   of   electromagnetic   radiation   by
controlling the distance accurately between the
5   point of evaporation and the tip of the straight
micropipette or fiber or the cantilever of the
bent   micropipette   or   fiber   and   this   distance
control is related to the diameter of the central
rotator of all the tips so that the cotan of the
10   angle   between   the   axis   of   the   straight
micropipette or fiber or the cantilever of the
bent micropipette or fiber approaches the cos of
this same angle.

14.   A device for the coating of tapered
15   straight and cantilevered structures for maximal
throughput   of   electromagnetic   radiation   by
controlling the angle as in claim 12 together with
the distance as in claim 13 to give most accurate
control of the final dimension of the resulting
20   aperture at the tip and controlling the thickness
of   the   coating   for   maximum   throughput   of
electromagnetic radiation

15.   A device in which the force sensing
capabilities of the probes as described in claim
25   1 are optimized so that the dimension of the taper
of the glass is appropriately chosen in the region
where   the   flexibility   of   the   structure   is
monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
30   be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
of the tips to dynamic movements of the surface

and/or application of non-contact protocols in
measuring surface forces

16.   A device in which the force sensing
capabilities of the probes as described in claim
5   2 are optimized so that the dimension of the taper
of the glass is appropriately chosen in the region
where the flexibility of the structure is
monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
10   be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
of the tips to dynamic movements of the surface
and/or application. of non-contact protocols in
15   measuring surface forces

17.   A device in which the force sensing
capabilities of the probes as described in claim
3 are optimized so that the dimension of the taper
of the glass is appropriately chosen in the region
20   where the flexibility of the structure is
monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
25   0.75 kHz and this allows both for maximal response
of the tips to dynamic movements of the surface
and/or application of non-contact protocols in
measuring surface forces

18.   A device in which the force sensing
30   capabilities of the probes as described in claim
4 are optimized so that the dimension of the taper
of the glass is appropriately chosen in the region

where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500

5   kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

10   19.   A device in which the force sensing capabilities of the probes as described in claim 5 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is

15   monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response

20   of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

          20.   A device in which the force sensing capabilities of the probes as described in claim

25   6 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can

30   be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface

and/or application of non-contact protocols in measuring surface forces

21.   A device in which the force sensing capabilities of the probes as described in claim 7 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application. of non-contact protocols in measuring surface forces

22.   A device in which the force sensing capabilities of the probes as described in claim 8 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces

23.   A device in which the force sensing capabilities of the probes as described in claim 9 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region

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where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500

5   kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

10   24.   A device in which the force sensing capabilities of the probes as described in claim 10 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is

15   monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response

20   of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

         25.   A device in which the force sensing capabilities of the probes as described in claim

25   11 are optimized so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can

30   be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface

and/or application of non-contact protocols in measuring surface forces.

26. A method of tapering a glass structure produced from glass tubes by glass pulling
5   technologies to generate an optimized geometry for high transmission efficiency of electromagnetic radiation that is appropriately coated with metal that includes a tapered region which is above the cut-off for the transmission of this radiation
10 having a cone angle of 6-10° and a subsequent tapered section that has a much larger cone angle that can be as much as 45° such a structure being optimized for both high throughput light transmission and excellent capabilities for force
15 sensing as a result of having an ultrahigh resonance frequencies for example 500 kHz and a Q factor of 600 and can thus be used in a variety of force sensing protocols.

27. A method as in claim 26 using a glass
20 capillary

28. A method as in claim 26 using an optical fiber

29. A method as in claim 27 that produces a cantilevered structure by bending the tip so that
25 the structure is suitable for normal force sensing in a variety of attractive, repulsive and non-contact imaging modes.

30. A device as in claim 28 that produces a cantilevered structure by bending the tip so that
30 the structure is suitable for normal force sensing

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in a variety of attractive, repulsive and non-contact imaging modes.

31. A method as in claim 27 that produces a cantilevered structure optimized for delivery of nanoquantities of liquid in a controlled fashion so that chemistry with nanometric control can be performed and this is accomplished by pressure on the liquid column inside the tapered micropipette, by chemical manipulation of the hydrophillicity of the surface and the tip, by the nature of the tapered geometry of the tip and the angle of the cantilever relative to the tip in order to prevent the spreading of the liquid column on the surface on which nanochemistry is to be performed .

32. A method as in claim 31 for producing a structure that has resonance frequencies that will allow for intermittent contact operation and will effectively permit imposed resonances on the tip that will allow, in the case of a liquid chemical in the tip, for the column of liquid to be effectively and reproducibly broken.

33. A method as in claim 31 that also optimizes the structure for maximum force sensitivity.

34. A method as in claim 32 that also optimizes the structure for maximum force sensitivity.

35. A method as in claim 27 for producing a cantilevered structure optimized for ion conductance.

36. A method as in claim 35 for producing a structure that optimizes the transmission of light that can excite either directly or through an optical fiber threaded through the micropipette an

5   ion sensing dye at the tip of the micropipette in a sol-gel glass that is also simultaneously conductive to ions and is also capable of static and dynamic force sensing.

37. A method for the coating of tapered

10   straight and cantilevered structures so that thin films can be deposited on these glass structures by rotation about the axis of the straight glass tip and/or by accurately varying the angle of incidence between the source and the tip target.

15   38. A method for the coating of tapered straight and cantilevered structures for maximal throughput of electromagnetic radiation by controlling the distance accurately between the point of evaporation and the tip of the straight

20   micropipette or fiber or the cantilever of the bent micropipette or fiber and this distance control is related to the diameter of the central rotator of all the tips so that the cotan of the angle between the axis of the straight

25   micropipette or fiber or the cantilever of the bent micropipette or fiber approaches the cos of this same angle.

38. A method for the coating of tapered straight and cantilevered structures for maximal

30   throughput of electromagnetic radiation by controlling the angle as in claim 37 together with the distance as in claim 38 to give most accurate

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control of the final dimension of the resulting
aperture at the tip and controlling the thickness
of the coating for maximum throughput of
electromagnetic radiation.

5   40.   A method for the optimization of the
force sensing capabilities of the probes as
described in claim 26 so that the dimension of the
taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
10   monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
15   of the tips to dynamic movements of the surface
and/or application of non-contact protocols in
measuring surface forces.

           41.   A method for the optimization of the
force sensing capabilities of the probes as
20   described in claim 28 so that the dimension of the
taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
25   be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
of the tips to dynamic movements of the surface
and/or application of non-contact protocols in
30   measuring surface forces.

           42.   A method for optimization of the force
sensing capabilities of the probes as described in

claim 29 so that the dimension of the taper of the
glass is appropriately chosen in the region where
the flexibility of the structure is monitored and
this results in a resonance that is controlled in
5   frequency and this frequency, f, can be altered by
controlling this dimension up to 500 kHz with a
width of the resonance, Δf, that can be 0.75 kHz
and this allows both for maximal response of the
tips to dynamic movements of the surface and/or
10   application of non-contact protocols in measuring
surface forces.

43.   A method for optimization of the force
sensing capabilities of the probes as described in
claim 30 so that the dimension of the taper of the
15   glass is appropriately chosen in the region where
the flexibility of the structure is monitored and
this results in a resonance that is controlled in
frequency and this frequency, f, can be altered by
controlling this dimension up to 500 kHz with a
20   width of the resonance, Δf, that can be 0.75 kHz
and this allows both for maximal response of the
tips to dynamic movements of the surface and/or
application of non-contact protocols in measuring
surface forces.

25   44.   A method for the optimization of the
force sensing capabilities of the probes as
described in claim 31 so that the dimension of the
taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
30   monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be

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0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

5   45.   A method for optimization of the force sensing capabilities of the probes as described in claim 32 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and

10   this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the

15   tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

       46.   A method for the optimization of the force sensing capabilities of the probes as

20   described in claim 33 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is monitored and this results in a resonance that is controlled in frequency and this frequency, f, can

25   be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in

30   measuring surface forces.

       47.   A method for the optimization of the force sensing capabilities of the probes as

described in claim 34 so that the dimension of the
taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
monitored and this results in a resonance that is
5   controlled in frequency and this frequency, f, can
be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
of the tips to dynamic movements of the surface
10   and/or application of non-contact protocols in
measuring surface forces.

48.   A method for the optimization of the
force sensing capabilities of the probes as
described in claim 35 so that the dimension of the
15   taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
be altered by controlling this dimension up to 500
20   kHz with a width of the resonance, Δf, that can be
0.75 kHz and this allows both for maximal response
of the tips to dynamic movements of the surface
and/or application of non-contact protocols in
measuring surface forces.

25   49.   A method for the optimization of the
force sensing capabilities of the probes as
described in claim 36 so that the dimension of the
taper of the glass is appropriately chosen in the
region where the flexibility of the structure is
30   monitored and this results in a resonance that is
controlled in frequency and this frequency, f, can
be altered by controlling this dimension up to 500
kHz with a width of the resonance, Δf, that can be

0.75 kHz and this allows both for maximal response of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.

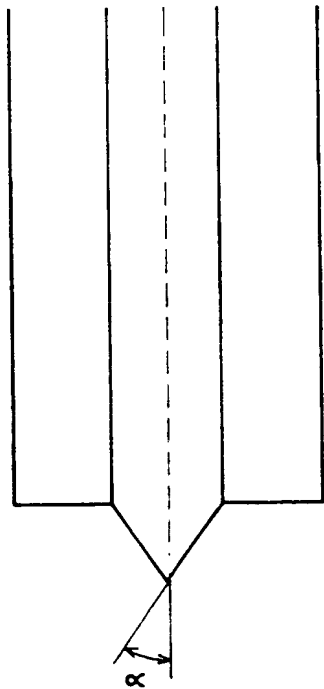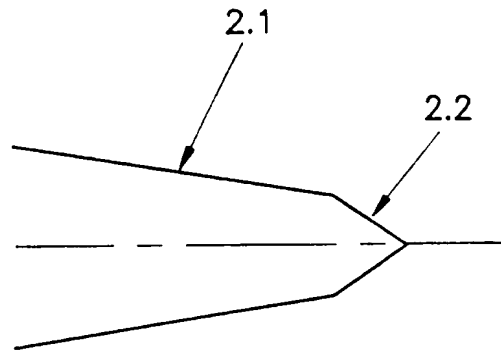5   50.   A method for the optimization of the force sensing capabilities of the probes as described in claim 37 so that the dimension of the taper of the glass is appropriately chosen in the region where the flexibility of the structure is
10   monitored and this results in a resonance that is controlled in frequency and this frequency, f, can be altered by controlling this dimension up to 500 kHz with a width of the resonance, Δf, that can be 0.75 kHz and this allows both for maximal response
15   of the tips to dynamic movements of the surface and/or application of non-contact protocols in measuring surface forces.
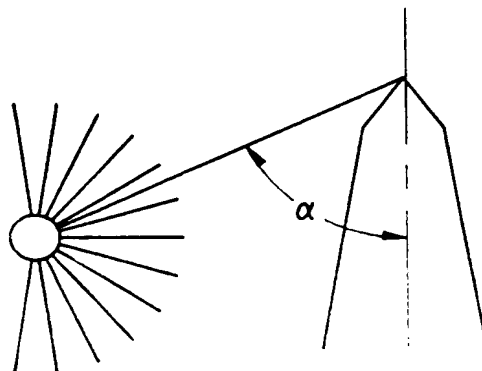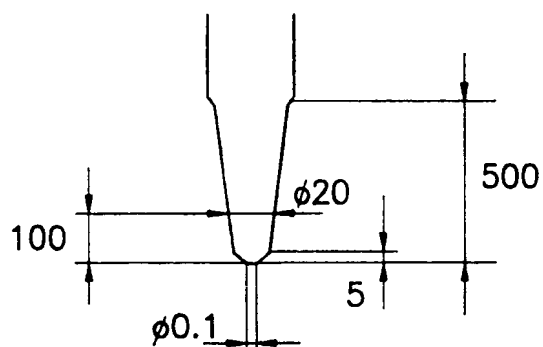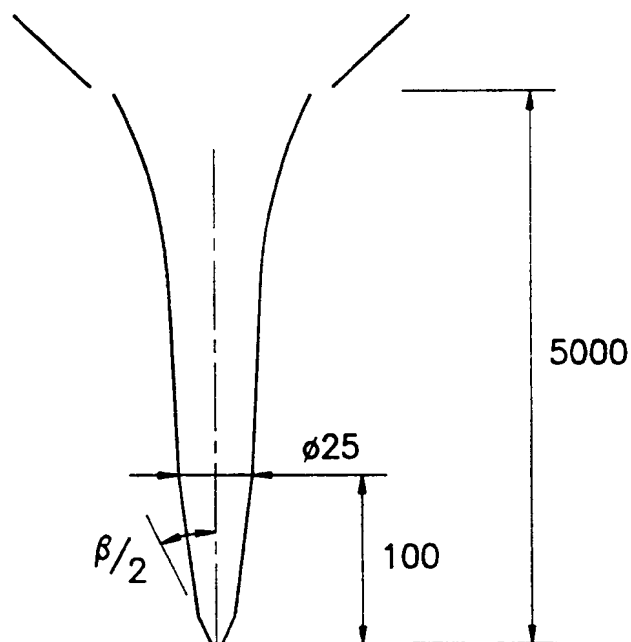
FIG. 1

FIG. 2

FIG. 3

SUBSTITUTE SHEET (RULE 26)

All dimensions are in micrometers

FIG. 4a

All dimensions are in micrometers
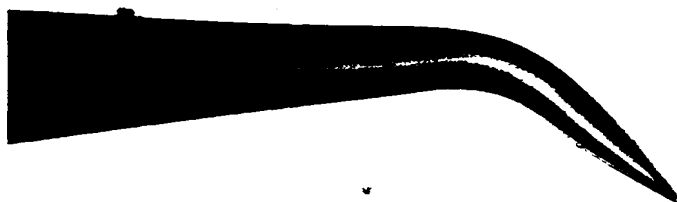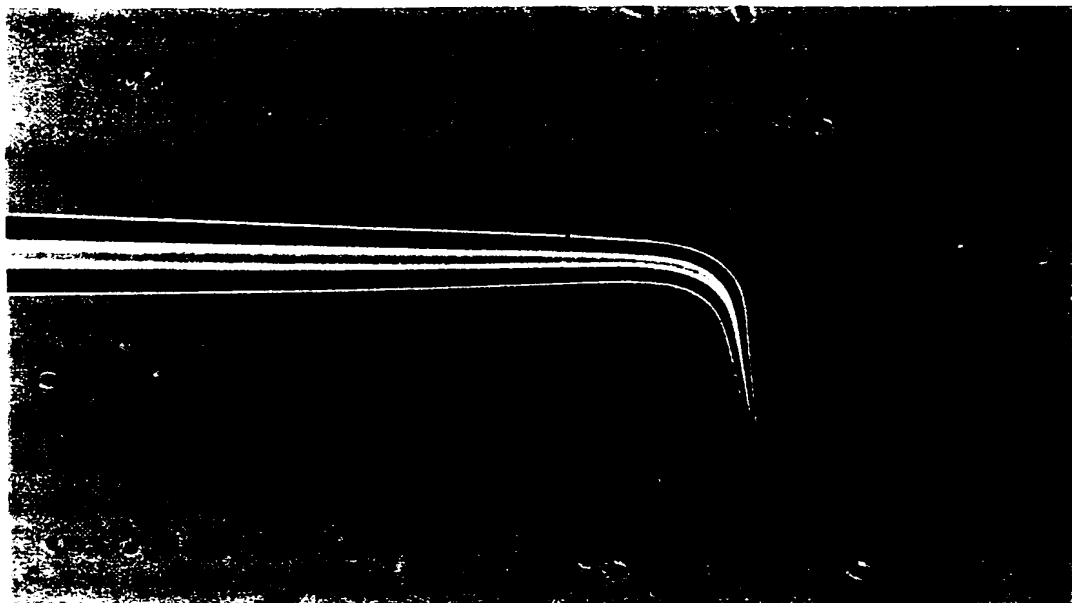
FIG. 4b

SUBSTITUTE SHEET (RULE 26)

FIG. 5

FIG. 6

FIG. 7

5 / 5

8.3

8.4

8.2

8.5

8.6

8.8   8.9   8.1   8.10 8.11

8.7

COATING SOURCE

VACUUM BELL JAR

α

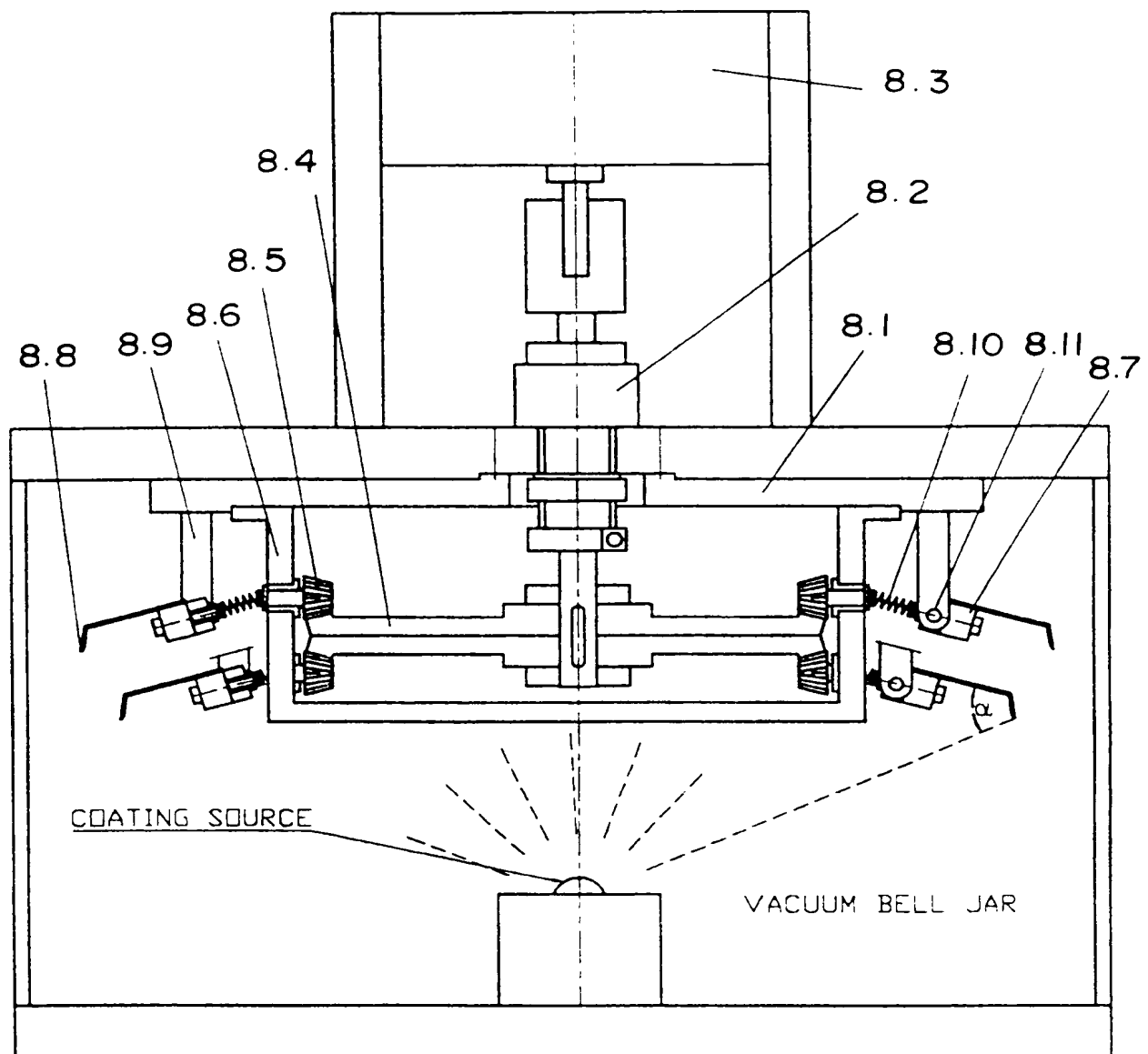FIG. 8

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US98/01577

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6)   :G02B 6/26
US CL   :385/43, 147, 38, 12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. :   385/43, 147, 38, 12, 15,

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

USPTO data base APS. Search terms: glass, tube, optical fiber, tapered, sensing.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,877,300 A (NEWHOUSE ET AL) 31 OCTOBER 1989 (31/10/89), see entire document.	1-50
A	US 4,915,467 A (BERKEY) 10 APRIL 1990 (10/04/90), see entire document.	1-50
A	US 5,044,716 A (BERKEY) 03 SEPTEMBER 1991 (03/09/91), see entire document.	1-50

☐ Further documents are listed in the continuation of Box C.   ☐ See patent family annex.

*	Special categories of cited documents:	
"A"	document defining the general state of the art which is not considered to be of particular relevance	
"E"	earlier document published on or after the international filing date	
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	
"O"	document referring to an oral disclosure, use, exhibition or other means	
"P"	document published prior to the international filing date but later than the priority date claimed	

"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&"	document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
20 MAY 1998	1 7 AUG 1998

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